National Institute of Standards & Technology

Report of Investigation

Reference Material 8441a

Wheat Hardness

This Reference Material (RM) is intended primarily for use in calibrating instruments used for the determination of hardness of bulk or single-kernel wheat. RM 8441a was prepared and analyzed by the Federal Grain Inspection Service (FGIS) program, Grain Inspection Packers and Stockyards Administration (GIPSA) of the United States Department of Agriculture (USDA). The USDA is the sole authority for all of the information provided in this report, including reference values and other technical information. One unit of RM 8441a consists of fifty pouches, five pouches each of five hard wheats and five soft wheats. Each pouch contains 20 g of material.

Reference Concentration Values: Reference values for hardness are provided in Tables 1 and 2. Reference values are noncertified values that are the best estimate of the true values based on available data; however, the values do not meet the National Institute of Standards and Technology (NIST) criteria for certification [1] and are provided with associated uncertainties that may reflect only measurement reproducibility or may not include all sources of uncertainty.

Expiration of Value Assignment: The value assignment of this RM is valid until **01 April 2011**, within the measurement uncertainties specified, provided the RM is handled and stored in accordance with the instructions given in this certificate. Value assignment is nullified if the RM is damaged, contaminated, or modified.

Maintenance of RM Value Assignment: The FGIS will monitor representative samples from each of the lots of wheat comprising this RM. If substantive technical changes occur that affect the value assignment before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

INSTRUCTIONS FOR USE

Each pouch should be allowed to warm to room temperature (23 °C ± 2 °C) immediately before use. If the RM is used to calibrate instruments for bulk hardness measurements, the wheat should be ground and used within 8 h after grinding.

RM 8441a was characterized by the USDA FGIS. The cleaning, dividing, packaging, and characterization of each wheat sample were performed at the FGIS under the direction of A.C. Johnson.

The technical aspects involved in the preparation and issuance of this Reference Material were coordinated by K.E. Sharpless of the NIST Analytical Chemistry Division.

Statistical consultation was provided by J. Yen of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this Reference Material were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

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NOTICE AND WARNING TO USERS

Storage: This RM, in unopened pouches, should be stored at 2 °C to 5 °C.

Warning: For laboratory use only. Not for human or animal consumption.

PREPARATION AND ANALYSIS[1]

Sources and Preparation of Materials: A set of wheat samples was designated by FGIS in 1996 as wheat hardness reference samples (WHRS). This set of samples is made up of ten separate lots of wheat (three pure varieties of hard red winter, two pure varieties of hard red spring, two pure varieties of soft red winter, and three pure varieties of soft white wheat) purchased from commercial sources. All lots were cleaned with a Carter-Day dockage tester and split into 15.9 kg (35 lb) sub lots using a Gamet rotator divider. The samples were stored in 19-liter (5-gallon) pails that were sealed with lids and stored at 2 °C to 5 °C. A sub lot of each of the WHRS was taken from storage and split into 20-gram portions using a Boerner divider and these sub samples were packaged in barrier-film aluminized pouches. Such sub lots were used to prepare RM 8441, which was issued in 1997, and RM 8441a, issued in 2004.

Bulk and Single-Kernel Reference Values and Uncertainties: RM 8441 and RM 8441a were prepared as transfer standards for linking the measurements of Near-Infrared Reflectance (NIR) instruments and Single-Kernel Characterization System (SKCS) 4100 instruments[1] to the National FGIS wheat hardness reference scale. Bulk hardness values assigned to the WHRS are the values that were obtained for these samples using two near-infrared reflectance instruments that had been sloped and biased to the standard wheat hardness samples. These hardness values were obtained from ten sets of measurements that were made on each WHRS over five weeks (two sets of measurements per week). Each set of measurements consists of determining the hardness values for five ground portions of a sample in two NIR instruments. The bulk hardness value assigned to the WHRS is the grand average of the 100 hardness measurements that were made on that sample (Table 1). The uncertainty in measuring the bulk hardness value of the WHRS was estimated from the standard deviation among the averages of twenty sets of mean values obtained for a sample (ten sets of means per NIR instrument). The single-kernel hardness values assigned to the WHRS are the grand averages of the hardness values that were found for these samples using 13 single-kernel characterization system instruments (SKCS 4100). The SKCS instruments were calibrated to give a mean hardness reading of 72.6 for the five hard WHRS and they were calibrated to give a mean hardness of 31.2 for the five soft WHRS. Each WHRS was measured in each of the 13 SKCS instruments in triplicate. The standard deviation of the 13 instruments' mean hardness values for a particular WHRS was taken as the uncertainty in the hardness values assigned to that WHRS (Table 2).

The hardness values reported in this document for RM 8441a are the hardness values that were assigned to the WHRS samples in 1996 (issued in 1997). To verify that the bulk and single-kernel hardness values of the WHRS had not changed in RM 8441a, bulk NIR hardness measurements and single-kernel SKCS 4100 hardness measurements were made on ten sets of RM 8441a. The NIR instrument was sloped and biased such that the mean of the hardness results for the hard RM 8441a was 76.7 and the mean of the hardness results for the soft RM 8441a was 30.5. The SKCS instrument was sloped and biased such that the mean of the hardness results for the hard RM 8441a was 72.6 and the mean of the hardness results for the soft RM 8441a was 31.2. Ten sets of three replicates of each of the 10 RM 8441a wheats were tested for hardness values using both of these instruments.

The grand average of the means of the hardness values for the bulk NIR hardness measurements and for the single-kernel SKCS 4100 hardness measurements for a particular RM 8441a material was calculated. In both cases, the bulk hardness and single-kernel hardness values found for RM 8441a were within the acceptance limits (i.e., the value found deviated from the assigned value by less than three times the uncertainty values listed in Tables 1 and 2) of the corresponding hardness values that were assigned to those samples in 1996. Therefore, the bulk NIR and single hardness values that are assigned to the RM 8441a material in this document (Tables 1 and 2) are values that were originally assigned to these samples in 1996.

[1]Certain commercial materials and equipment are identified in order to adequately specify the experimental procedure. Such identification does not imply a recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment are necessarily the best available for this purpose.

The mean hardness values listed in Table 1 are the values that the user should obtain using an NIR instrument standardized to RM 8441a using AACC Method 39-70A [2], such that the mean hardness value measured for the five hard RM wheats (M_{H1} to M_{H5}) is 76.7 and the mean of the hardness measured for the five soft RM wheats (M_{S1} to M_{S5}) is 30.5. On a properly standardized NIR instrument, using AACC Method 39-70A, the bulk hardness value measured for each wheat should be within three times the uncertainty values ($3 \times u_c$) listed in Table 1.

The wheat hardness values listed in Table 2 are the values that the user should obtain using an SKCS 4100 instrument that has been sloped and biased to RM 8441a, following the instructions in the SKCS 4100 Operation Manual [3], such that the mean of the hardness values measured for the five hard RM wheats (M_{H1} to M_{H5}) is 72.6 and the mean of the hardness scores measured for the five soft RM wheats (M_{S1} to M_{S5}) is 31.2. On a properly sloped and biased SKCS 4100 instrument, the hardness value measured for each wheat should be within three times the uncertainty values ($3 \times u_c$) listed in Table 2.

Table 1. Mean Bulk NIR Hardness Values for RM 8441a Wheats
(NIR Instrument; AACC Method 39-70A)

Wheat	Hardness Score	Uncertainty (u_c)
Hard-1 (M_{H1})	74.7	1.9
Hard-2 (M_{H2})	75.8	1.9
Hard-3 (M_{H3})	63.7	1.6
Hard-4 (M_{H4})	77.5	2.1
Hard-5 (M_{H5})	91.8	2.8
Soft-1 (M_{S1})	30.0	0.7
Soft-2 (M_{S2})	29.9	1.3
Soft-3 (M_{S3})	31.1	0.9
Soft-4 (M_{S4})	29.8	1.1
Soft-5 (M_{S5})	31.5	1.5

Table 2. Mean Single-Kernel Hardness Values for RM 8441a Wheats
(SKCS 4100 Instrument)

Wheat	Hardness Score	Uncertainty (u_c)
Hard-1 (M_{H1})	79.0	1.1
Hard-2 (M_{H2})	66.3	0.9
Hard-3 (M_{H3})	68.5	1.0
Hard-4 (M_{H4})	63.5	1.2
Hard-5 (M_{H5})	85.5	0.8
Soft-1 (M_{S1})	24.7	0.5
Soft-2 (M_{S2})	26.1	0.7
Soft-3 (M_{S3})	34.4	0.6
Soft-4 (M_{S4})	34.2	0.6
Soft-5 (M_{S5})	36.6	0.9

General Comments: The bulk and single-kernel hardness methods give different values for the individual reference wheats and for M_H and M_S. This is because two very different techniques are used to determine wheat hardness. The NIR instruments are responding to differences in the particle size of the ground material while the SKCS instruments depend on crushing force, moisture content, mass, and size per kernel. The computational procedure for NIR and SKCS 4100 standardization using RM 8441a is provided in the appendix of this Report of Investigation.

If RM 8441a is used for standardizing instruments other than NIR and the SKCS 4100 instruments, the hardness values measured may be different from the values listed in Tables 1 and 2.

Comments and inquiries from users of RM 8441a will be welcome and should be directed to:

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REFERENCES

[1] May, W.; Parris, R.;, Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136, U.S. Government Printing Office: Washington, DC (2000).

[2] AACC 39-70A; American Association of Cereal Chemists: Approved Methods AACC, 16th ed; St. Paul, MN (1995).

[3] Instruction Manual, *Single-Kernel Characterization System, Model SKCS 4100*; Perten Instruments, Inc.: 6444 South Sixth Street Road, Springfield, IL 62707.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; *email srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.*

APPENDIX A

Computation Procedure for the Standardization of NIR and SKCS 4100 Instruments Using RM 8441a.

The process by which the mean of the hardness values measured for the five hard RM 8441a wheats is adjusted to the fixed value, H_B, and the mean of the hardness values measured for the five soft RM 8441a wheats is adjusted to the fixed value, S_B is called standardization. For bulk hardness measurements, H_B and S_B are fixed at 76.7 and 30.5, respectively. For single-kernel hardness measurements, H_B and S_B are fixed at 72.6 and 31.2, respectively.

Let M_{H1}, M_{H2}, M_{H3}, M_{H4}, and M_{H5} be the means of the hardness values for two or more hardness measurements performed on Hard 1, Hard 2, Hard 3, Hard 4, and Hard 5 RM wheats, respectively, and let M_{S1}, M_{S2}, M_{S3}, M_{S4}, and M_{S5} be the means of the hardness values for two or more hardness measurements performed on Soft 1, Soft 2, Soft 3, Soft 4, and Soft 5 RM wheats, respectively.

Then $M_H = (M_{H1} + M_{H2} + M_{H3} + M_{H4} + M_{H5}) \div 5$

and $M_S = (M_{S1} + M_{S2} + M_{S3} + M_{S4} + M_{S5}) \div 5.$

An NIR or SKCS 4100 instrument is standardized to RM 8441a if $M_H = H_B$ and $M_S = H_S$. If $M_H \neq H_B$ and/or $M_S \neq H_S$, then the hardness values measured for the RM using these instruments are sloped and biased such that $M_H = H_B$ and $M_S = H_S$.

The following relationship holds true for H_B, S_B, M_H, and M_S: $H_B = a + (b \times M_H)$ and $S_B = a + (b \times M_S)$, where, a and b are the bias and slope factors used to adjust the measured hardness values to the appropriate fixed values.

The slope factors can be computed as follows: $b = (H_B - S_B) \div (M_H - M_S)$.

The bias factor can be computed from $a = H_B - (b \times M_H)$ or $a = S_B - (b \times M_S)$.

Therefore, the standard hardness value (SHV) for each wheat can be determined using the following relationship: SHV = a + (b × old HV) where old HV is the hardness measured for RM 8441a.

Example: Suppose that the SKCS 4100 yielded the hardness values listed in Table A1 for RM 8441a.

Table A1. Example Hardness Values Measured for RM 8441a Using the SKCS 4100

	Replicate			
Sample	R1	R2	R3	Mean
Hard-1 (M_{H1})	76.5	76.4	76.2	76.4
Hard-2 (M_{H2})	65.0	63.9	63.5	64.1
Hard-3 (M_{H3})	64.4	64.7	67.1	65.4
Hard-4 (M_{H4})	61.3	60.5	61.0	60.9
Hard-5 (M_{H5})	82.3	82.7	83.4	82.8
Soft-1 (M_{S1})	24.4	23.4	23.4	23.7
Soft-2 (M_{S2})	25.3	24.2	24.1	24.5
Soft-3 (M_{S3})	32.3	33.1	32.5	32.6
Soft-4 (M_{S4})	32.6	33.4	31.6	32.5
Soft-5 (M_{S5})	33.7	34.7	34.6	34.3

$M_H = (M_{H1} + M_{H2} + M_{H3} + M_{H4} + M_{H5}) \div 5$
$= (76.4 + 64.1 + 65.4 + 60.9 + 82.8) \div 5$
$= 69.9$

$M_S = (M_{S1} + M_{S2} + M_{S3} + M_{S4} + M_{S5}) \div 5$
$= (23.7 + 24.5 + 32.6 + 32.5 + 34.3) \div 5$
$= 29.5.$

For SKCS 4100, $H_B = 72.6$ and $S_B = 31.2$

$b = (H_B - S_B) \div (M_H - M_S)$
$= (72.6 - 31.2) \div (69.9 - 29.5)$
$= 41.4 \div 40.4$
$= 1.02475$

$a = S_B - (b \times M_S)$
$= 31.2 - (1.02475 \times 29.5)$
$= 1.0.$

SHV = a + (b × old HV)

so that

M_H = a + (b × old HVM_H)
$= 1.0 + (1.02475 \times 69.9)$
$= 72.6$

and

$M_S = a + (b \times \text{old } HVM_S)$
$= 1.0 + (1.02475 \times 29.5)$
$= 31.2.$

Also,

SHV (Hard 1) = a + (b × old HV Hard 1)
= 1.0 + (1.02475 × 76.4)
= 79.2

and

SHV (Soft 1) = a + (b × old HV Soft 1)
= 1.0 + (1.02475 × 23.7)
= 25.2

and so on.